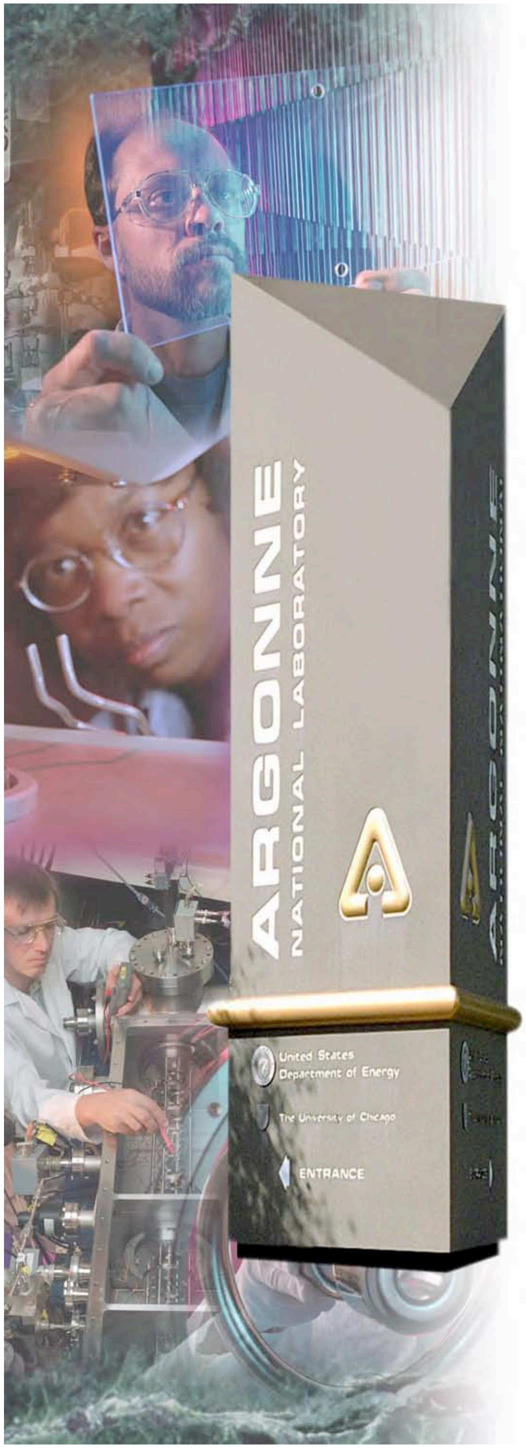

Lithium thin-film coatings studies for NSTX

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Plasma Facing Components (PFC) Meeting
Livermore, CA
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Argonne National Laboratory

Office of Science
U.S. Department of Energy

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Office of Science Laboratory
Operated by The University of Chicago

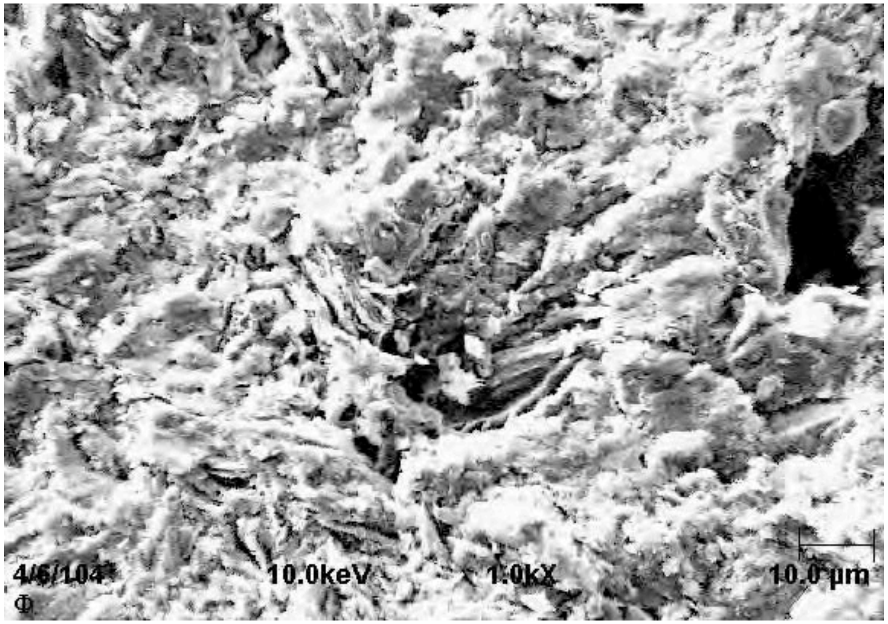
Outline of talk

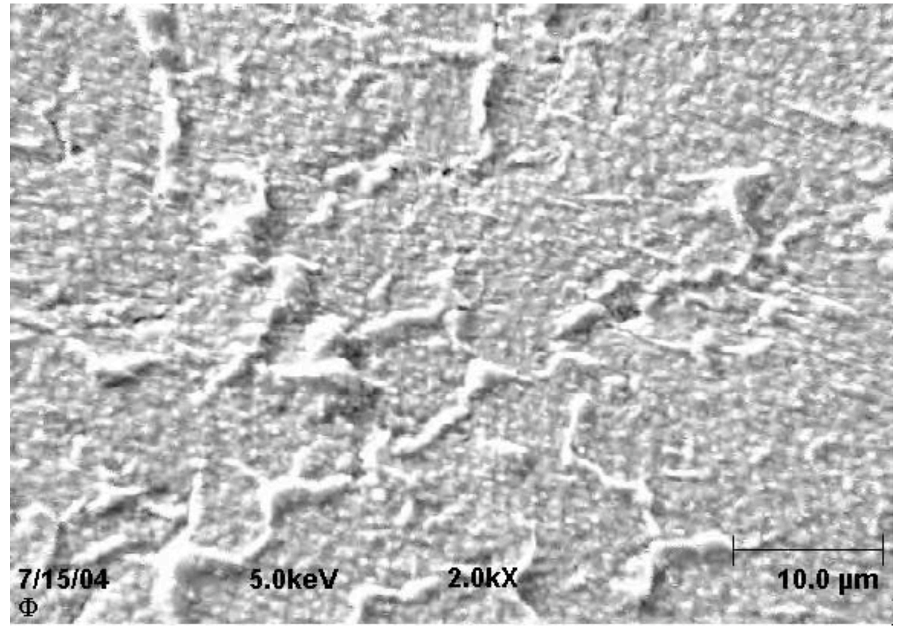
- Overview of work with lithium thin-film coatings on graphite
- Description of *IMPACT* and *ARIES* facilities
- Behavior of evaporated Li on polished and non-polished ATJ graphite
- The role of boron coatings on Li/C interactions
- Analysis of NSTX tile with exposure to Li pellet injections (tile provided by: H. Kugel, et al.)
- Preliminary work on Li/Mo and Li/W systems
- Summary
- Future work

Overview of Li thin-film coatings

- **Lithium thin-film coatings on graphite tiles are proposed as Phase 1 of Module A plan in NSTX.**
  - Phase 2 of Module A will consist of W or Mo-coated surfaces to be used with lithium
- **Experiments at Argonne and Sandia/Livermore study thin-film lithium-based systems with respect to:**
  - Study of lithium evolution on graphite surfaces and boron-treated graphite surfaces (polished and unpolished)
  - Study of impurity evolution (H,N,O) on lithium-treated graphite surfaces also as function of temperature and background water pressure
  - Bombardment-induced sputtering (He^+, D^+, etc…) of lithium-treated graphite and alternate substrates
  - Evolution of lithium on Mo or W substrates

Sample preparation

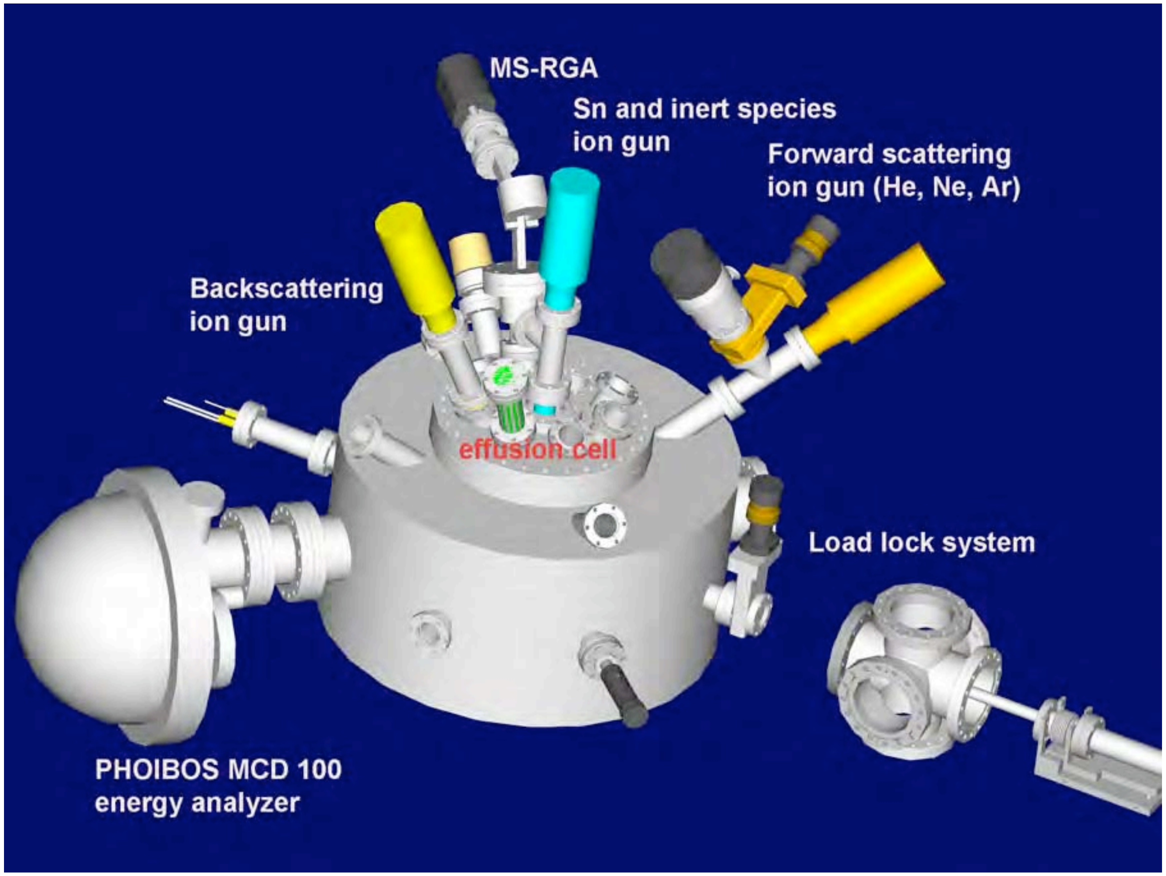




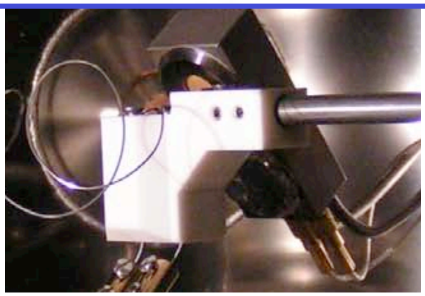
- **Samples consisted of non-polished (left SEM image) and polished/annealed (right SEM image) ATJ graphite samples.**
- **A 300 nm Li coating was evaporated on both types of ATJ graphite**
- **A 300 nm Li/ 70 nm B stack was prepared on both types of ATJ graphite**
- **For this study we looked at Li/B/C and Li/C on polished ATJ graphite and Li/B/C on unpolished ATJ graphite**
- **Additional studies on 300 nm Li evaporated on 10 µm W and Mo samples**

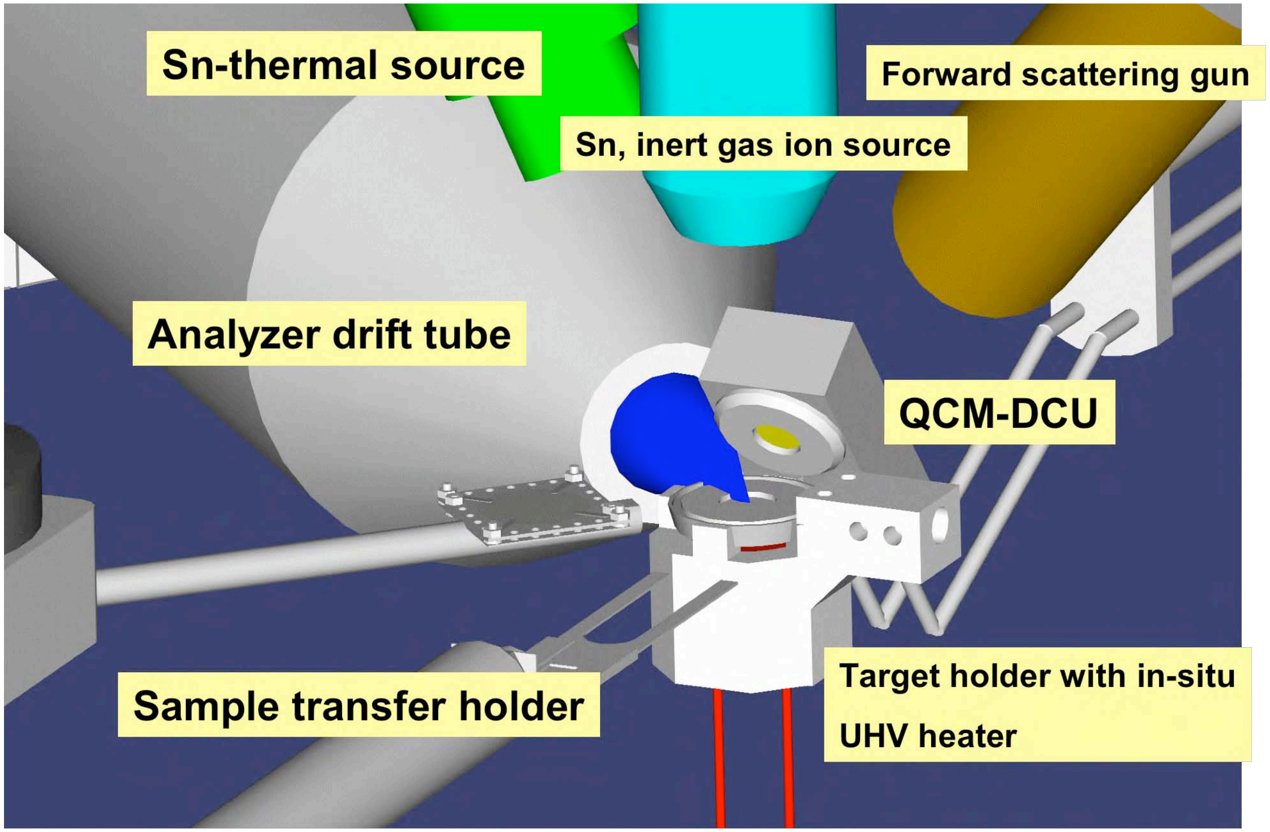
IMPACT Experiment Setup

- Singly-charged particle source providing flux range from 10^{11}-10^{17} ions/cm^2/sec and Energy: 10-5000 eV.
- Xe, Li and Sn ion sources as well as: He,Ne,Ar, Kr, O, H, D, N
- In-situ rotatable Faraday cup for beam profile analysis.
- E-beam evaporator: Sn and Li atom sources (alternate EUV radiators)
- In-situ heater (25-500 ºC) on rotary manipulator for angle-of-incidence studies.



IMPACT In-situ metrology

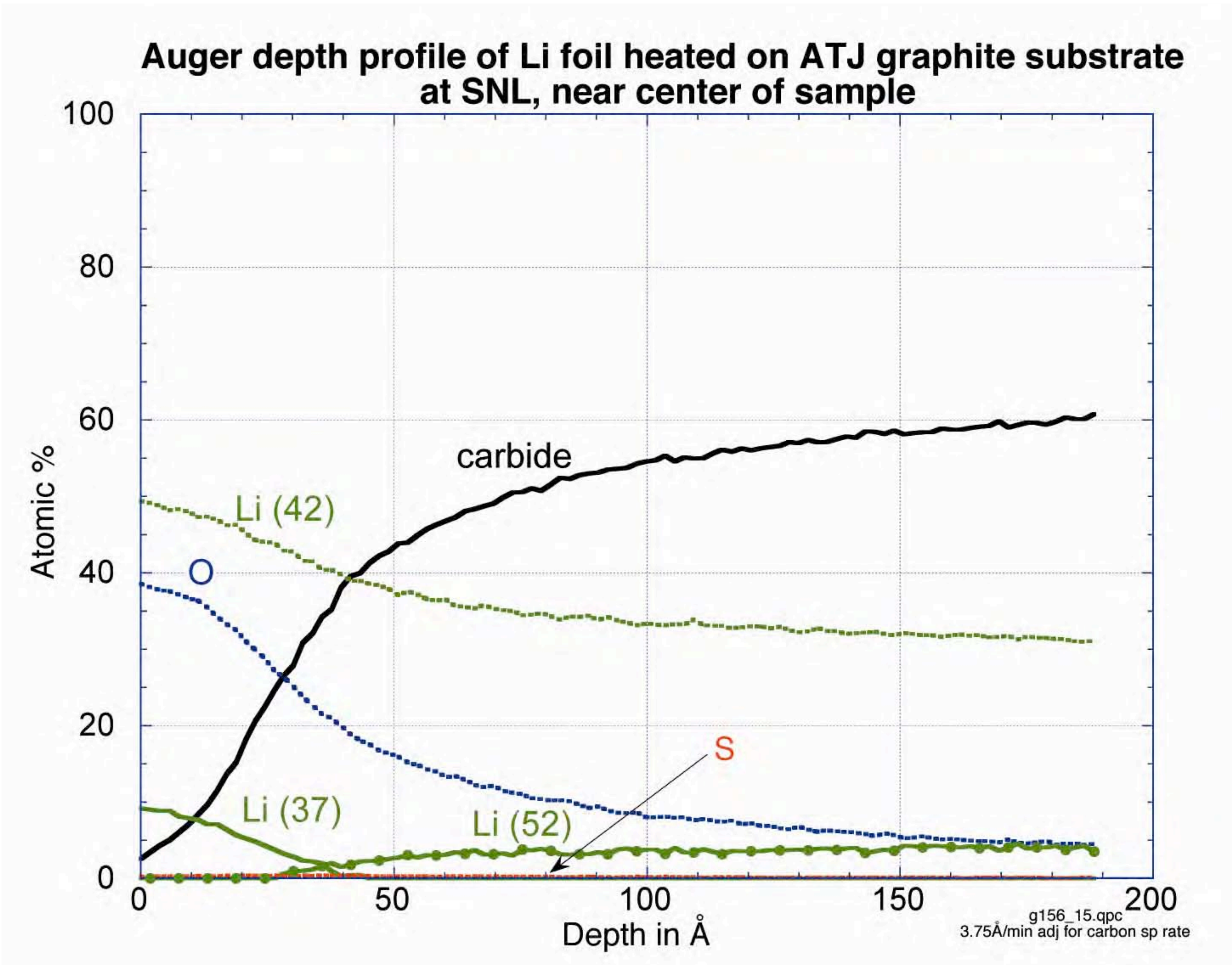




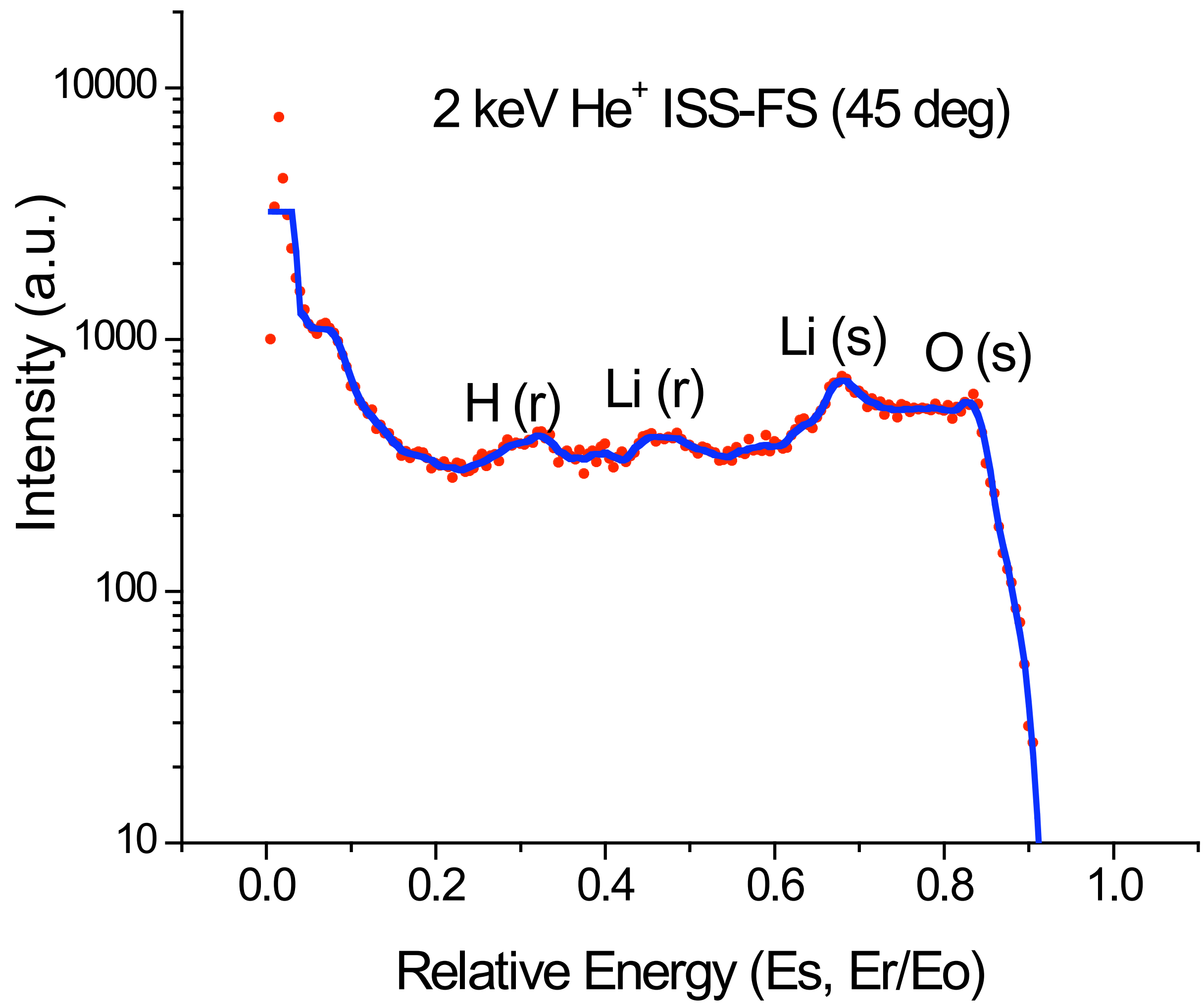
- Dual quartz crystal microbalance for total erosion measurements.
- **In-situ Metrology:** Low-energy ion scattering spectroscopy (forward and backward scattering), direct recoil spectroscopy, Auger electron spectroscopy, X-ray photoelectron spectroscopy using a SPECS 100-MCD.

Angle-resolved ion energy spectrometer (ARIES) at SNL/CA

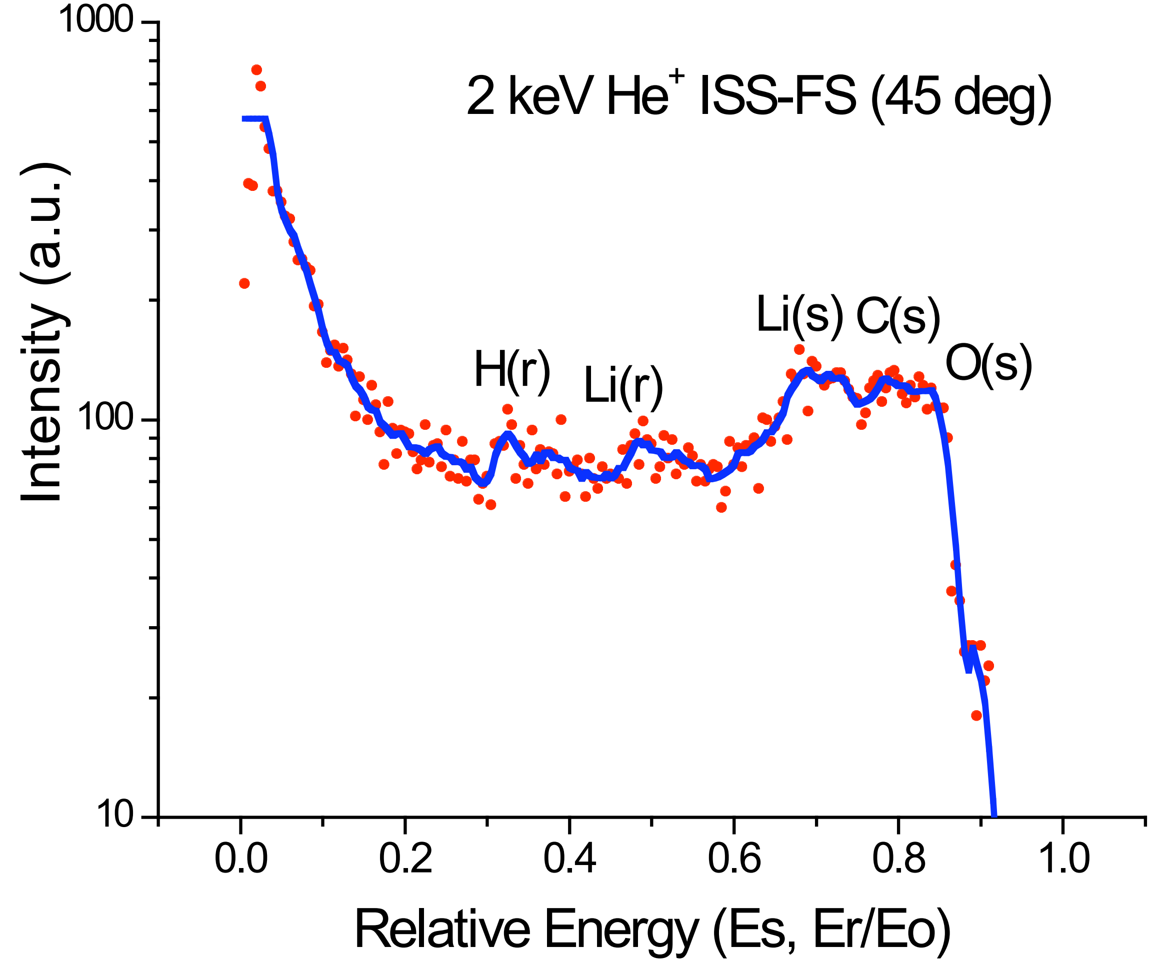
- **Experiment designed to provide detailed top atomic layer analysis of thin films and materials (angularly-resolved low-energy ion scattering spectroscopy and Auger electron spectroscopy)**
- **The system has two sections:**
  - A low-energy (0.1 – 3 keV) ion source differentially pumped chamber
  - UHV analysis chamber (P ~ 10^{-10} Torr)
- **ATJ graphite samples polished and annealed were prepared with various coatings.**
- **In addition, W and Mo substrates were also coated with 300 nm Li and selected samples were delivered to ANL.**
- **ISS, AES and SEM analysis of samples.**


Auger depth profile of Li foil heated on ATJ graphite substrate
at SNL, near center of sample
Atomic %
100
80
60
40
20
0
carbide
Li (42)
O
Li (37)
Li (52)
S
0
50
100
150
200
Depth in Å
g156_15.qpc
3.75Å/min adj for carbon sp rate

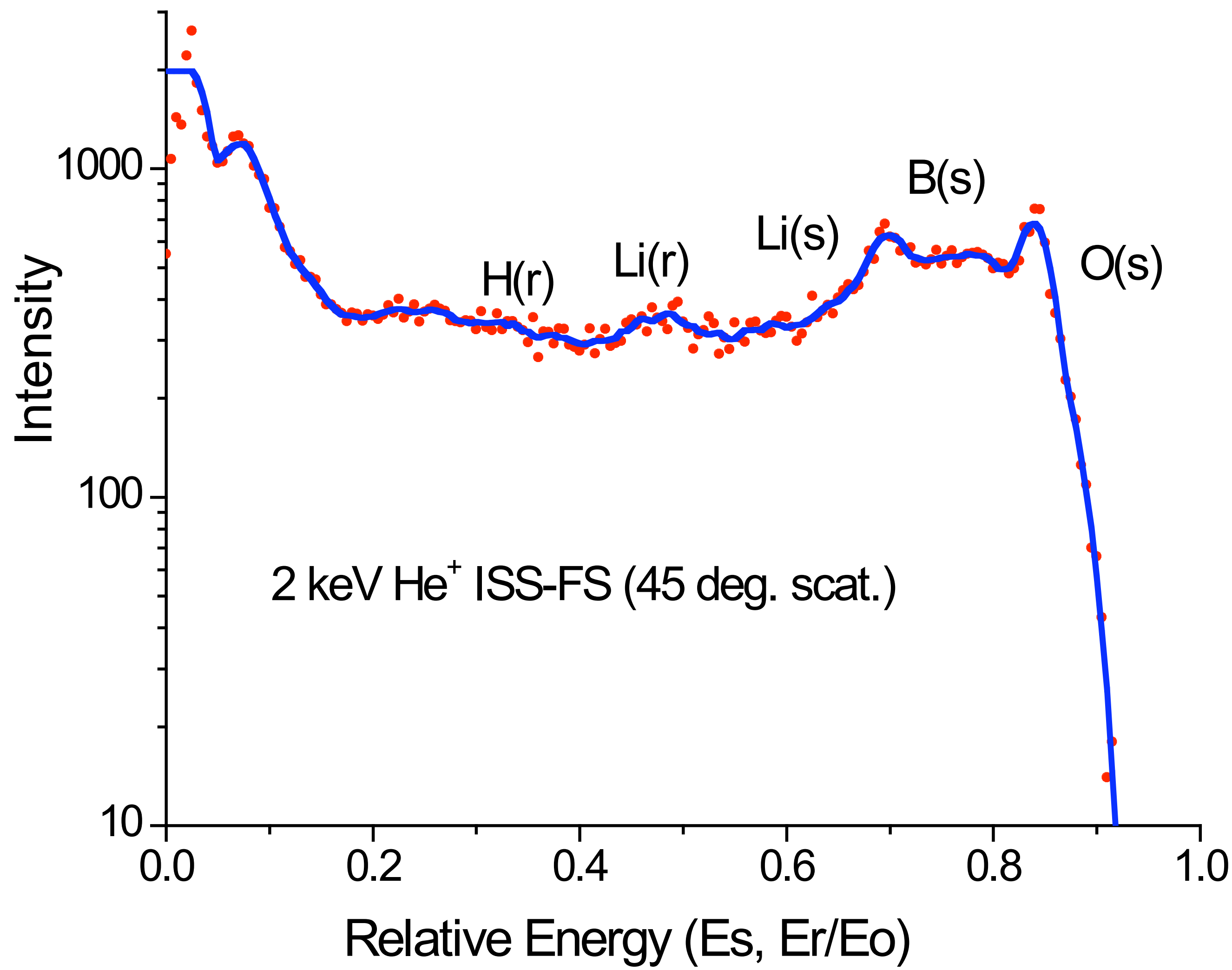
Evaporated Li on polished graphite at 25 ºC



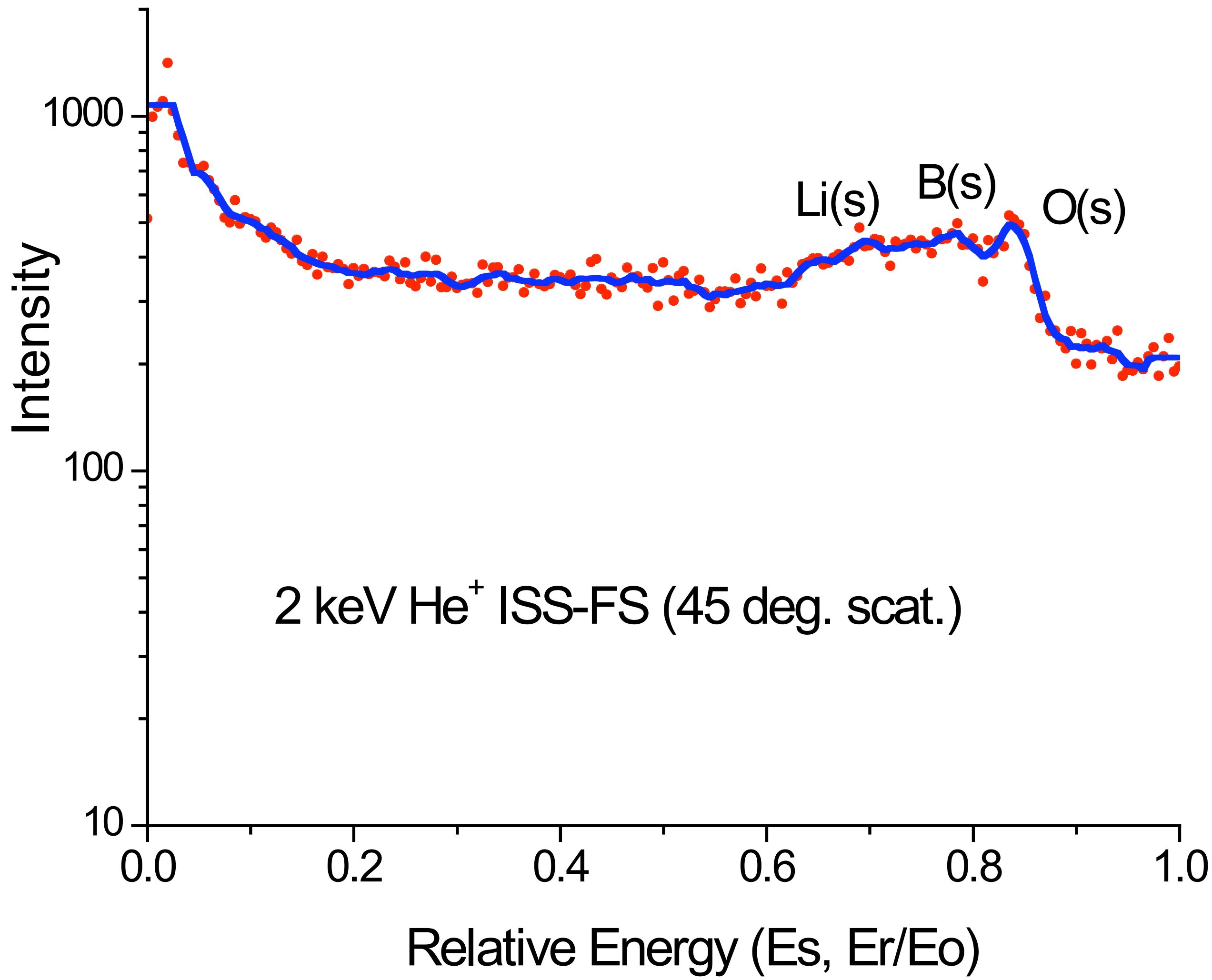
Evaporated Li on polished graphite at 300 ºC



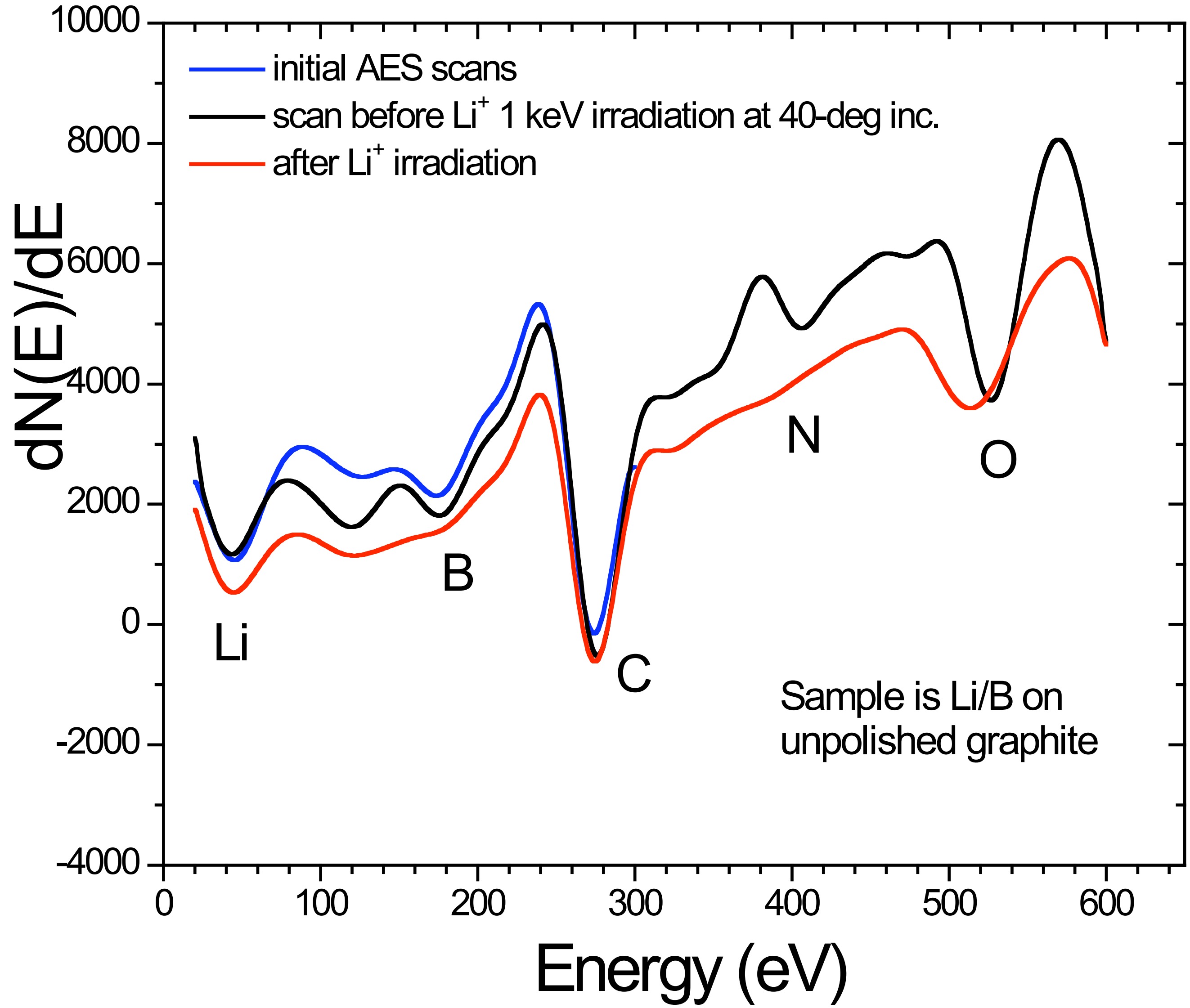
Li/B on polished graphite at 25°C



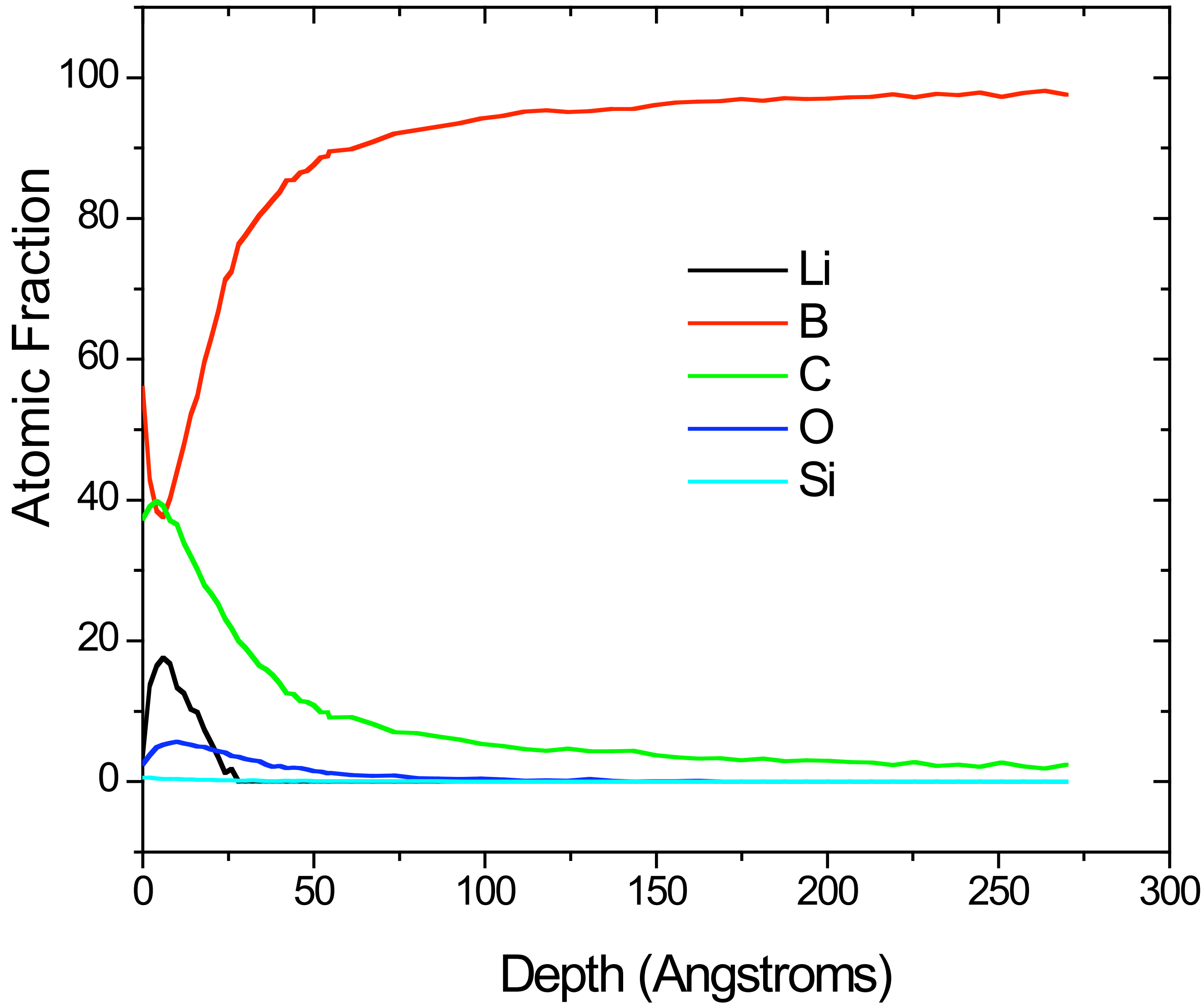
Li/B on polished graphite at 300°C



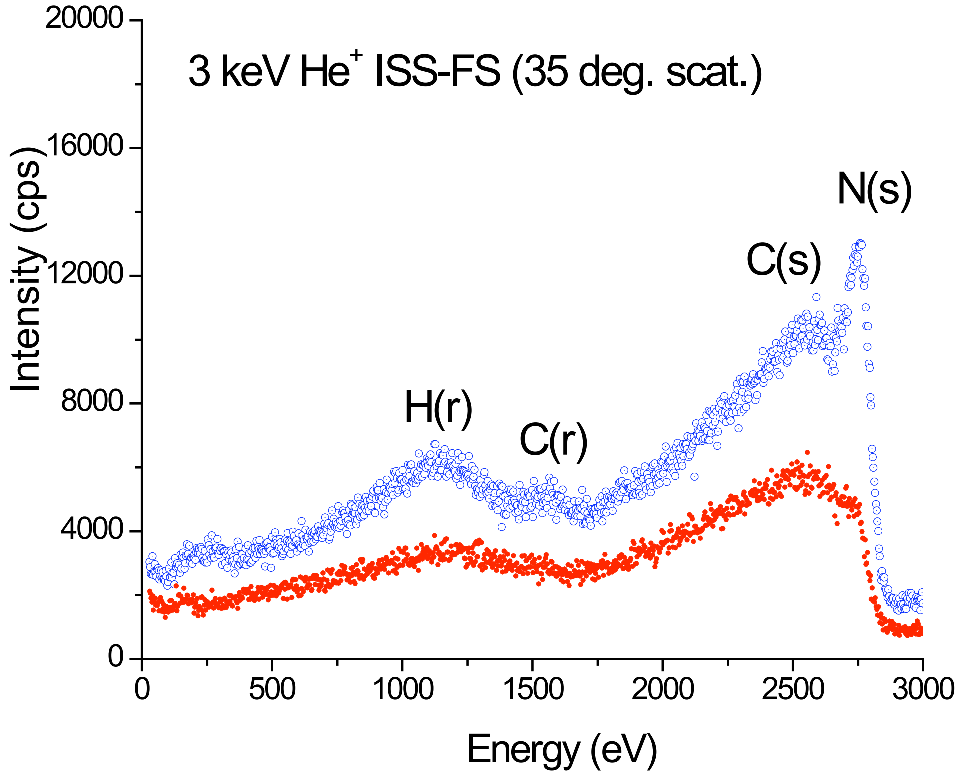
Li/B on unpolished graphite

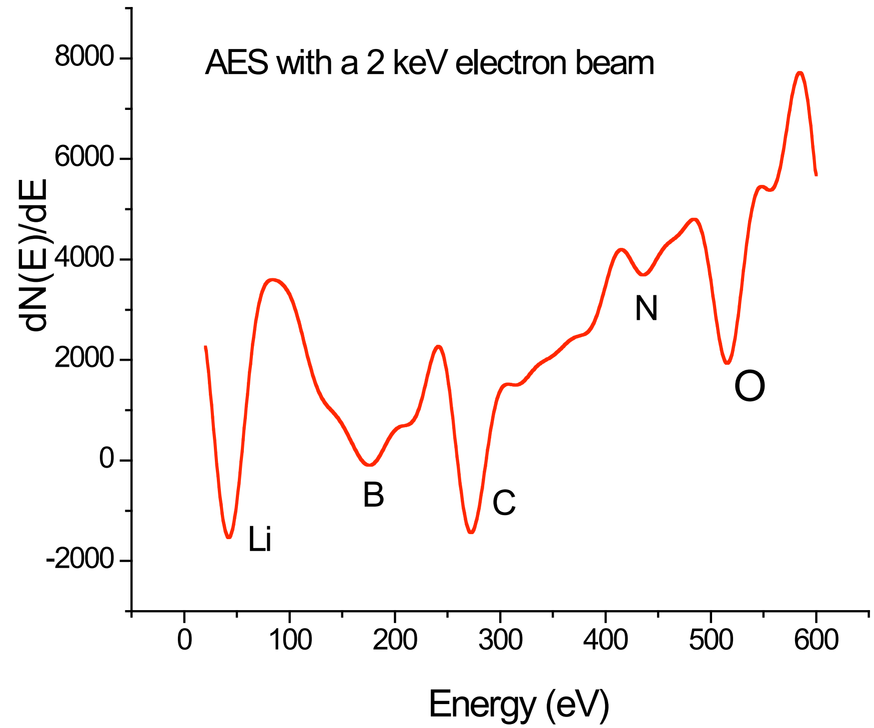


The role of B on Li deposition on C



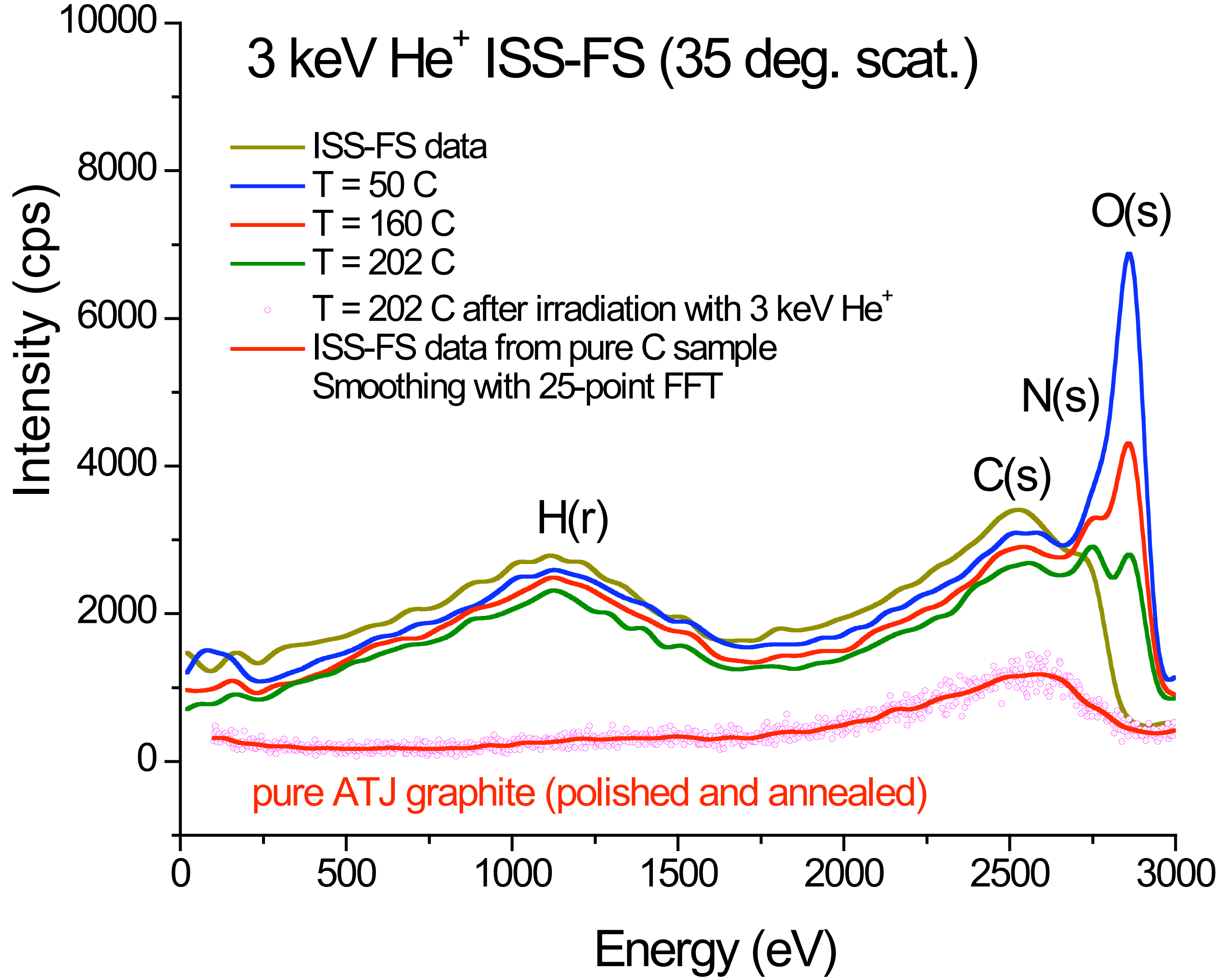
Li/B on polished graphite (R.T.)





- **The presence of N is evident in both the in-situ ISS and AES.**
- **After exposure to 1 μA of 2 keV He^+, the N peak in the ISS is gone.**

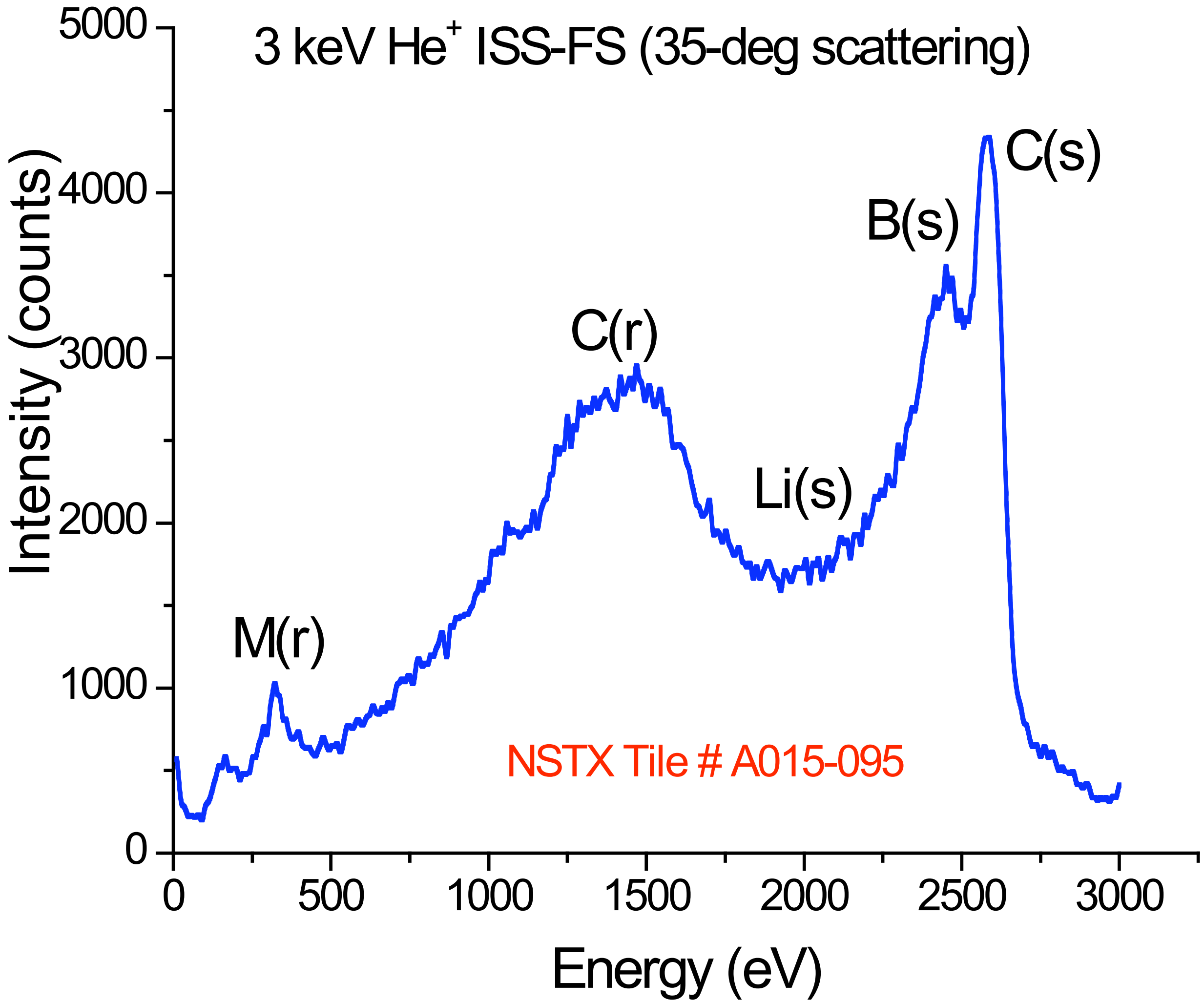
Li/B on polished graphite (cont.)



Case: heater outgasses, trapped H_2O breaks up on complex mixed surface

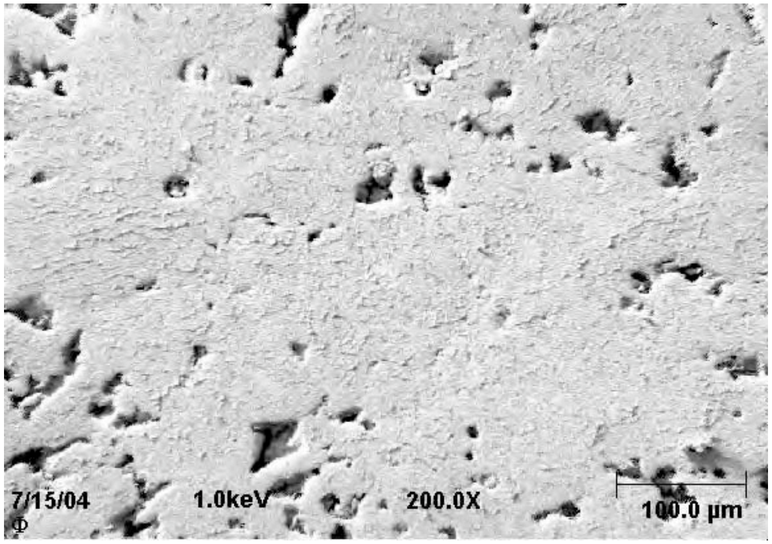
Pioneering Science and Technology

Li on boron-treated graphite tiles in NSTX

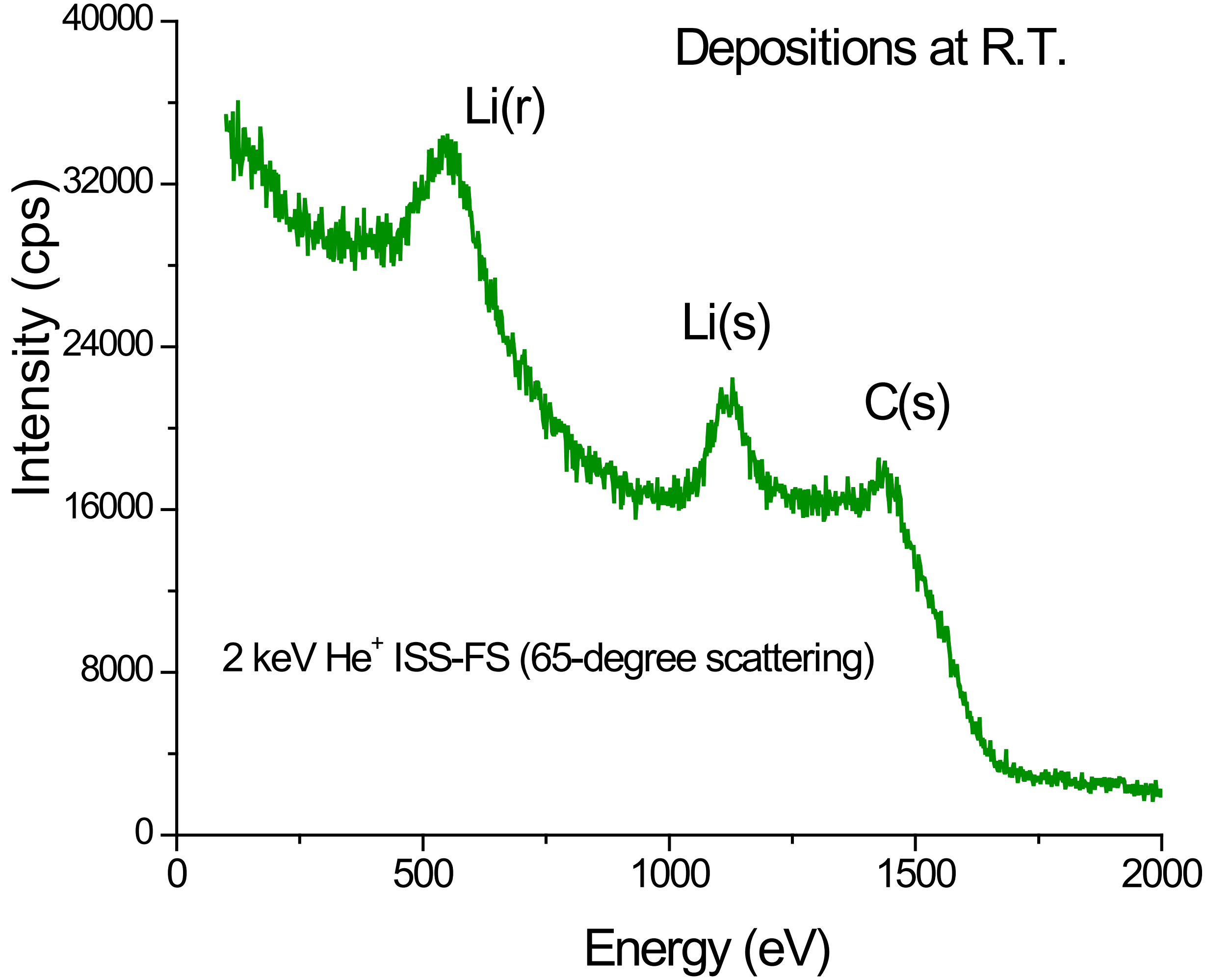


Role of boron in C on Li diffusion

- Li diffuses into C even in the presence of B and other impurities. Therefore B does not act as a barrier to incident Li.
- However, all samples shown were analyzed having a time lag between growth and analysis.
- In addition, a complex morphology and surface composition is found whereby O, N and H are quite active.
- Formation of Li compounds could also be formed.

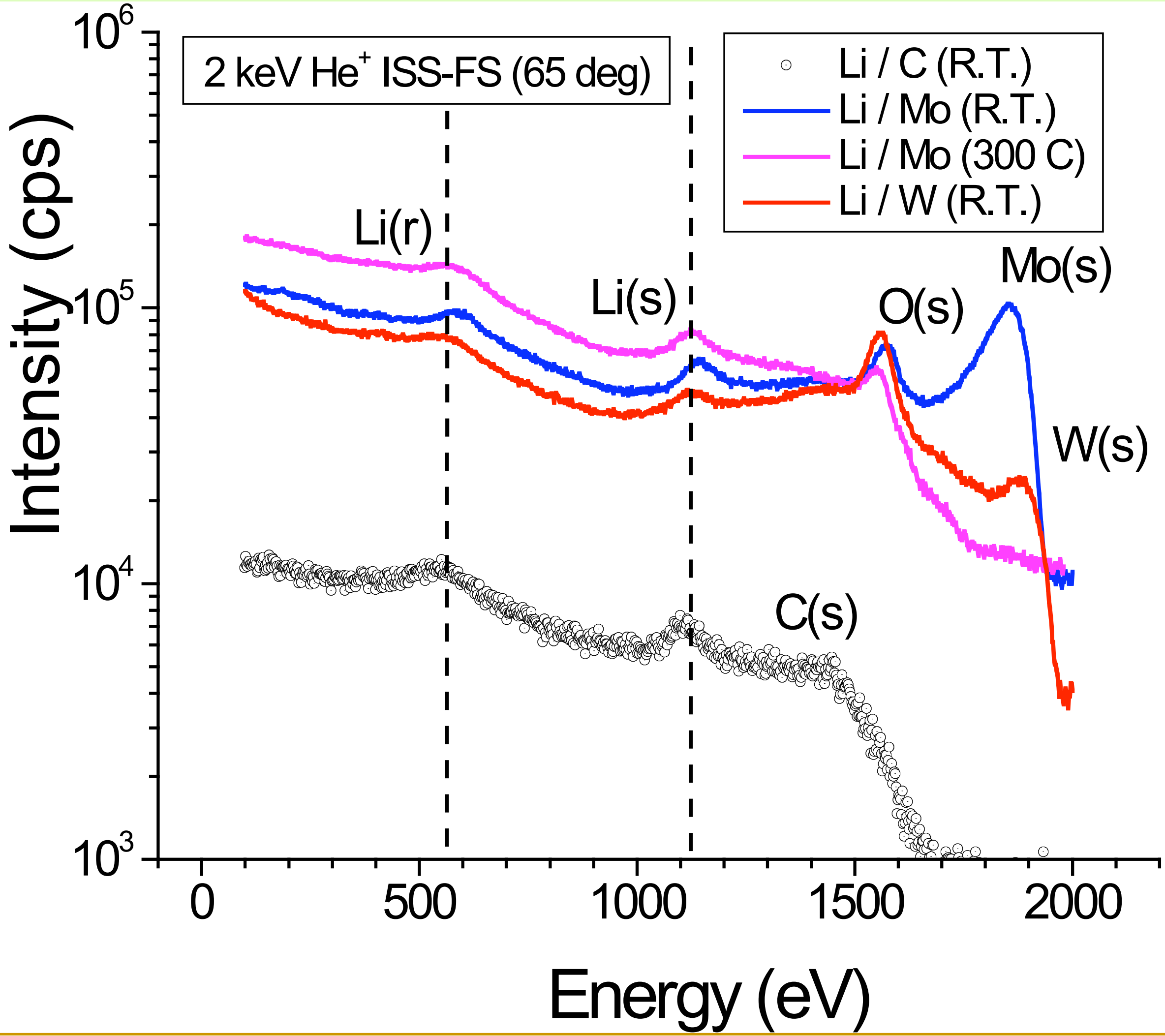


In-situ evaporation of Li on C

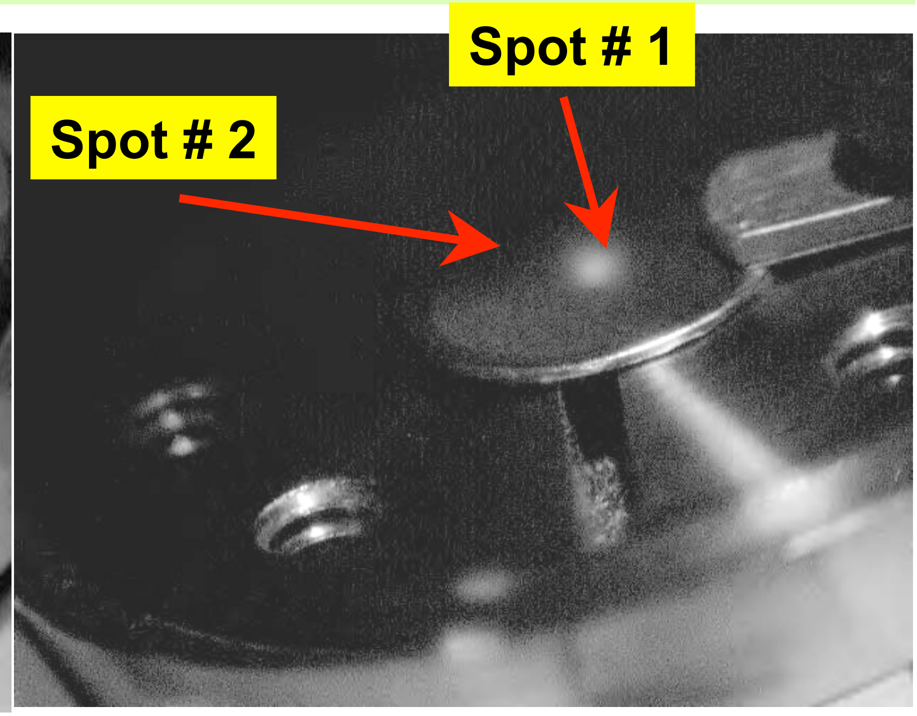


Real time measurement of Li deposition on ATJ graphite

Comparison to Li evaporation on other metal substrates (W, Mo)

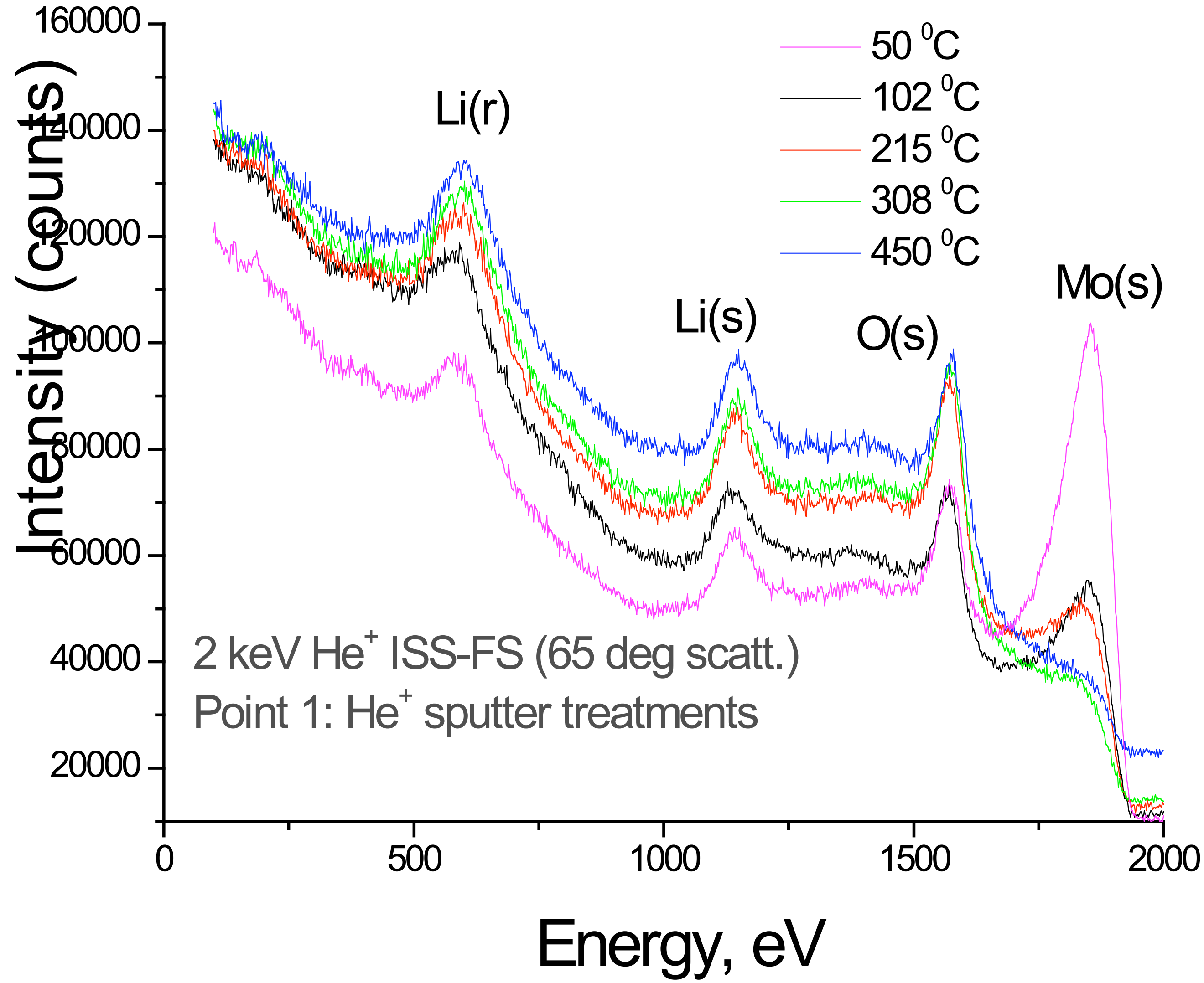


Preliminary work on Li/W and Li/Mo systems



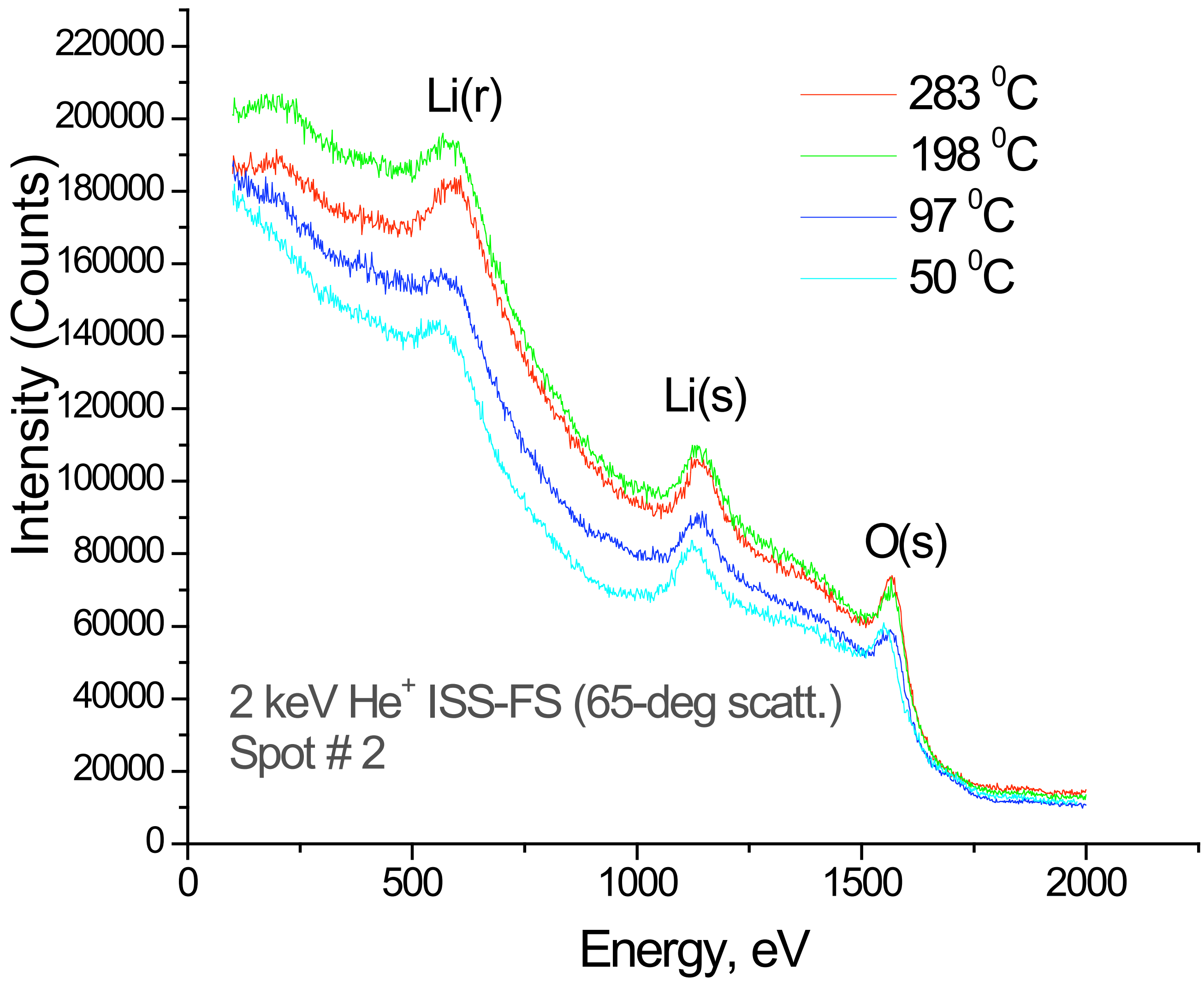
- **A Watec CCD camera on a Edmund Sci lens provides in-situ visual inspection of the sample during irradiations.**
- **In fact, ion-induced fluorescence allows us to “see” the He^{+} beam and be able to tune and move it using the manipulator arm**

Evaporated Li layer evolution on Mo



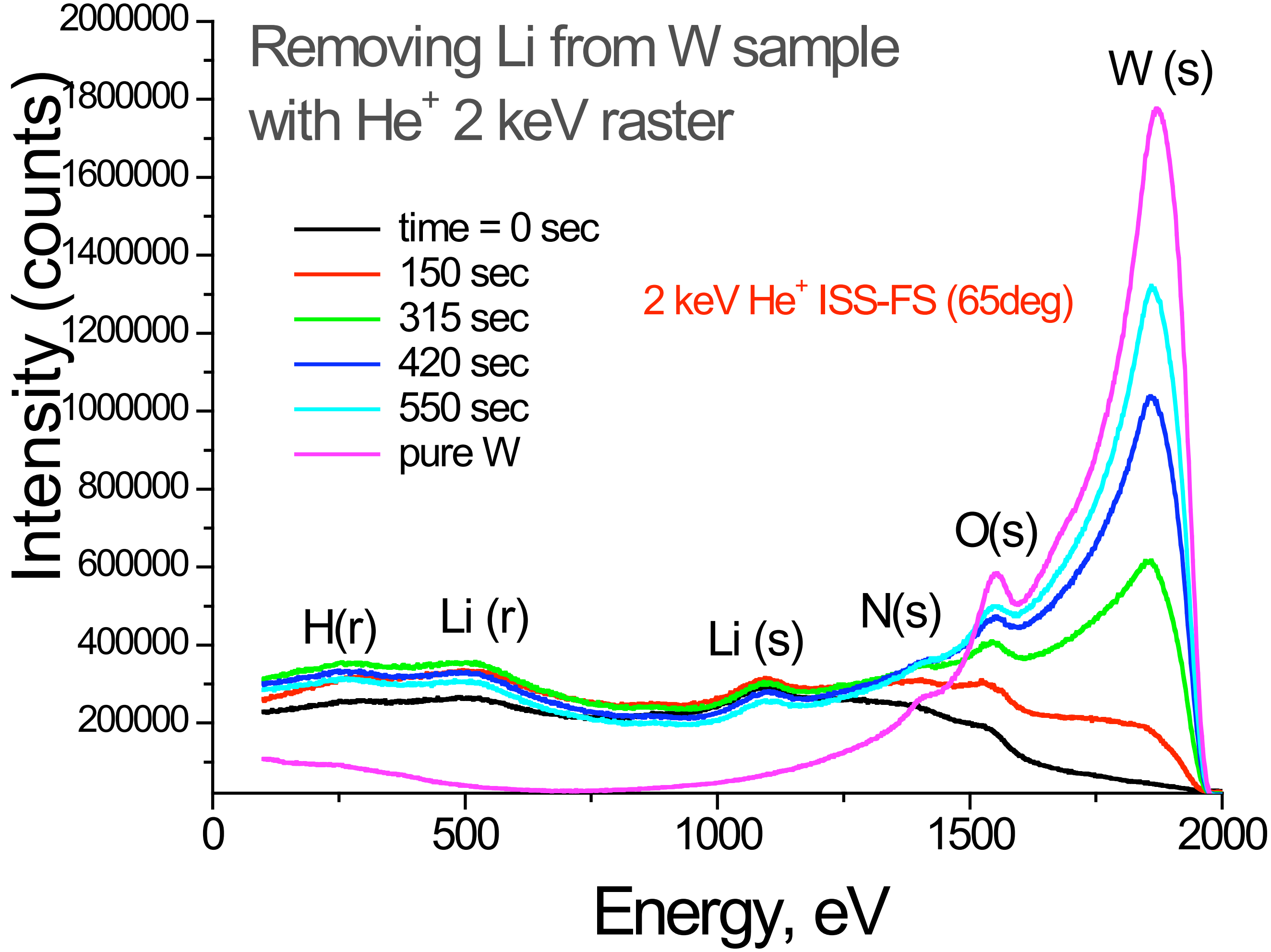
Spot # 1

Evaporated Li layer evolution on Mo

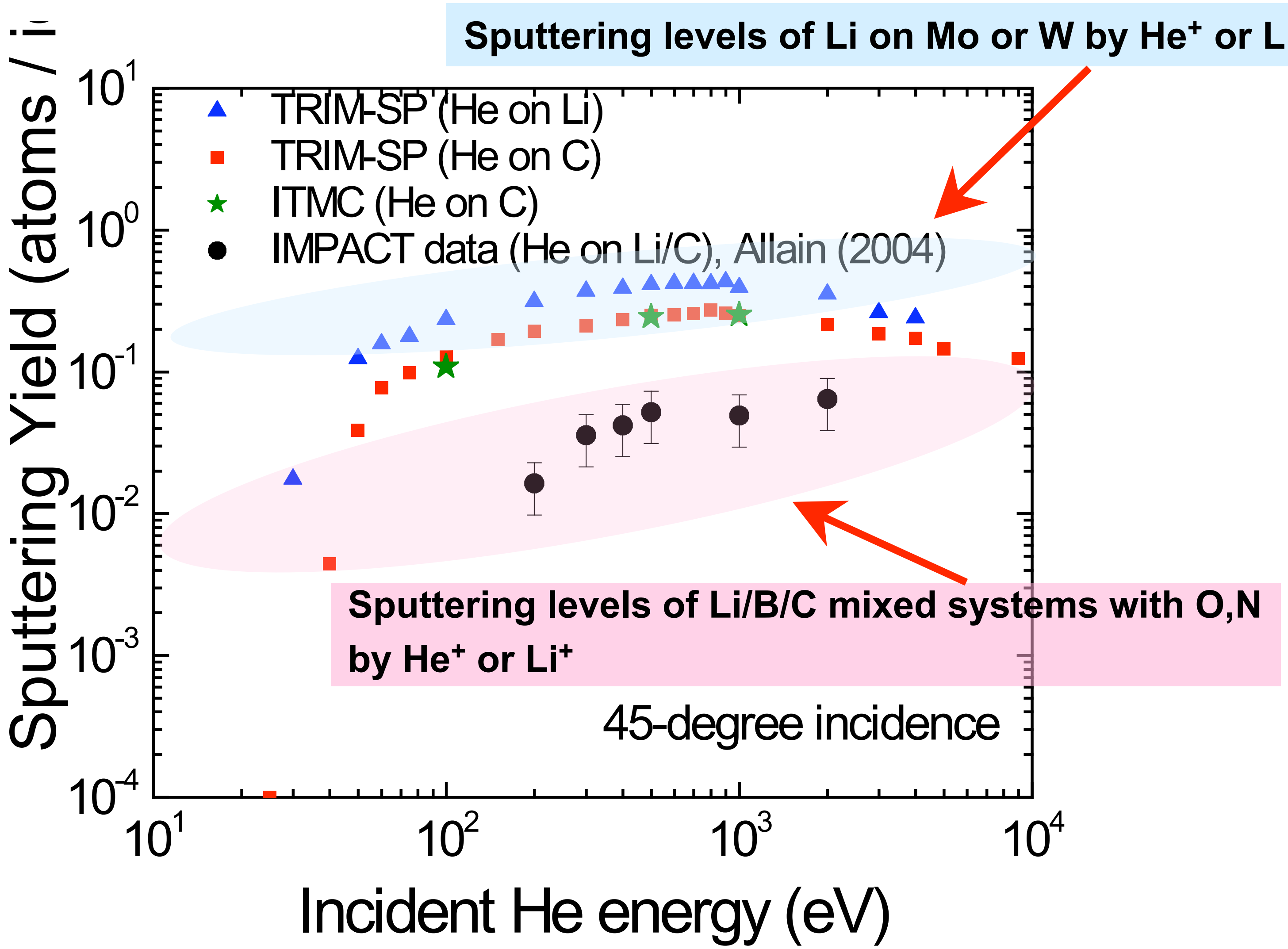


Spot # 2

Removal of Li layer from W



He⁺and Li⁺ erosion of ATJ graphite



Summary

- A complex mixed surface is measured in both the IMPACT and ARIES facilities.
- The presence of boron does not prevent Li from diffusion into graphite (in both polished and unpolished forms)
- Oxygen, nitrogen contents are increased at surface and subsurface regions by presence of B in BC_x systems.
- Preliminary real-time Li evaporation in-situ results in Li being observed at the surface of graphite.
- Li evaporation on W and Mo substrates show surface segregation as a function of temperature.
- Oxygen is found along with Li on all substrates.
- Erosion is severely reduced in the complex Li/B/C mixed system.

Future Work and Plans

- **Understand quantitatively lithium surface kinetics on graphite and alternate substrates (W, Mo)**
- **Help guide NSTX Li pellet injection experiments**
  - Li concentrations that would lead to favorable "low recycling" regime conditions
  - Role of oxygen on D-recycling
- **Next step is D^+ irradiation of graphitic surfaces with and without B presence. Compare to alternate substrates (W, Mo).**
- **Assess effect of mixing layers on secondary sputtered ion fraction of lithium atoms (~ 2/3 of sputtered lithium in the ionized state) and erosion reduction.**

Acknowledgements

- Perry Plotkin and William Klimowych, *PRIME* (Particle and Radiation Interaction with Matter Experiments) facility engineers at Argonne
- R. Kaita, R. Majeski and H. Kugel for helpful discussions and feedback
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